

SOP: Environics Series 9100 Calibration System

Revision: 2 (MM)

Date: October 25, 2000

STANDARD OPERATING PROCEDURE

FOR

**ROUTINE OPERATION OF THE ENVIRONICS
SERIES 9100 COMPUTERIZED AMBIENT
MONITORING CALIBRATION SYSTEM IN CRPAQS**

STI-999214

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October 25, 2000

SOP: Environics Series 9100 Calibration System

Revision: 2 (MM)

Date: October 25, 2000

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1. SCOPE AND APPLICABILITY

The Environics Series 9100 Computerized Ambient Monitoring Calibrator produces highly precise mixes of gases. These gas mixtures can be used to evaluate and optimize the performance of several gas monitors.

2. SUMMARY OF METHOD

The S-9100 was designed to precisely blend two or more gases with or without ozone. The instrument has a total of eight inlet ports. Each of these ports has a mass flow controller to set the amount of gas that flows through the line. The first port is fed purified air from a pure air generator. The instrument has two different modes of operation depending on if the ozone generator is being used or not.

The instrument has a built-in ozone generator, which serves two purposes: one to generate ozone and the other to generate ozone to react with NO to form NO₂. If the ozone generator is being used, part of the pure air stream is diverted into the generator. A known amount of ozone is generated by an ultraviolet lamp. If ozone is the desired end product, this stream is combined with a precise amount of pure air to achieve the desired concentration and sent to the outlet of the calibrator. If NO₂ is the desired end product, this stream is combined with a precise amount of NO gas from the second port. The gases are allowed to mix and react in a reaction chamber. Then, this stream is recombined with a precise amount of pure air to create the desired concentration and is sent to the outlet of the calibrator.

When the ozone generator is not being used, a precise amount of pure air enters through the first port and is mixed with a precise amount of another gas from the second port to create the desired concentration. This mixture is then sent to the outlet of the calibrator.

3. DEFINITIONS

OGC = Output Gas Concentration

MFC = Mass Flow Controller

NO = Nitric oxide

NO₂ = Nitrogen dioxide

O₃ = Ozone

DAS = Data Acquisition System

4. HEALTH & SAFETY WARNINGS

Some gases can be explosive or otherwise reactive when blended. The user must check gas compatibility before blending. Failure to observe these precautions may result in damage to the instrument, serious injury, or death.

Pressurized cylinders are extremely dangerous when mishandled. Proper regulators, use of safety caps, and proper restraints are mandatory. Avoid cross contamination when attaching regulators or making manifold connections. Always consult the gas supplier for proper safety procedures. Failure to observe these cautions may result in serious injury or death.

Always disconnect electrical power before servicing the instrument. Connect the unit to a grounded electrical outlet.

5. CAUTIONS

The pressure on the gas inlets of the instrument must not exceed 150 psig. The instrument requires 115 VAC power at 50/60 Hz. The instrument should not be operated in temperatures below 0° Celsius or above 50° Celsius.

6. INTERFERENCES

The purity and precision of the calibration gases output from the calibrator are dependent on the purity of the air and gases fed to the calibrator. Therefore, proper operation of the pure air generator must be maintained and a reliable supplier for the calibration gases must be used.

7. PERSONNEL QUALIFICATIONS

Safety training is essential when dealing with pressurized gas cylinders. Proper operation of the instrument requires a basic understanding of chemistry.

8. APPARATUS & MATERIALS

- Pure air is fed to the calibrator using 1/4" Teflon tubing and fittings.
- All other gases are fed to the calibrator using 1/8" stainless steel tubing and fittings.
- Teflon tubing and fittings are used to route the calibration gases from the calibrator to the calibration manifold.
- Teflon tubing, Teflon tee fittings, and Teflon solenoid valves are used to construct the calibration manifold. The calibration gases will be routed to the NO_y and O₃ instruments in the first phase of CRPAQS. The line from the calibrator will be split using a tee fitting. One outlet of the tee will be sent to a 3-way solenoid valve that feeds the ozone analyzer. The second outlet from the tee will be sent up to another tee. One outlet of this tee will be sent to a 2-way solenoid valve that feeds the NO_y analyzer. The second outlet of the tee will be sent to a 2-way solenoid valve that is used as a vent. This is a total of two tee fittings, two 2-way solenoid valves, and two 3-way solenoid valves.
- A 9/16" wrench, 7/16" wrench, and a small adjustable wrench are required to assemble the calibration system.

9. SITE & EQUIPMENT PREPARATION

- Off site equipment acceptance
 - The S-9100 should be removed from its shipping container and inspected for damage. Remove the top cover by removing the two small screws on each side of the top and inspect the interior of the instrument. Look for possible loose parts or visible damage. If there are any loose circuit boards, press down to reset them before proceeding to connect power. If there is no visible damage, replace the cover and proceed.
- Off site pre-deployment equipment test
 - Plug in the instrument, making sure that the supplied power is 115 VAC and 50/60 Hz.
 - Turn the power on using the front panel rocker switch. The display panel should run through a short self-testing procedure and then indicate READY. If READY appears on the screen, the user should turn the instrument off and begin connecting the gases. If there is no display, refer to the section on troubleshooting.
 - Zero air is attached to port 1 with 1/4" tubing and should be delivered at 35 psig.

▪ On-site equipment acceptance

- First, the same procedure should be followed on-site as was followed off-site.
- Then, all of the calibration gases should be attached to the calibrator using 1/8" stainless steel tubing. Each of the inlets is labeled with the gas that should be input.
- Next, the zero and span should be set on the ozone analyzer. Calibration sequences will be actuated by the DAS. There are several sequences pre-programmed into the DAS and are described in detail in the DAS SOP.
- After setting the zero and span values, the ozone analyzer should be calibrated. The sequence for the ozone analyzer consists of five steps, each step runs for 10 minutes. The concentrations for each step are 40, 80, 120, 160, and 200 ppb ozone.
- Next, a five point calibration must be performed on the NO_y analyzer. The sequence for the NO_y analyzer consists of five steps running for 20 minutes, with concentrations of 0, 20, 100, 200, and 450 ppb NO.

▪ On-site equipment installation

- The calibrator should be installed as close as possible to the several instruments it will be supplying with gases.

▪ On-site connection of equipment to the data acquisition system

- The RS-232 port will be used to remotely control the calibrator. Using the panel soft keys, enter the RS-232 menu. Make sure the settings for ports 1 and 2 are as follows:

On/Off	ON	OFF
Baud Rate	6400	6400
Terminal Type	CPU	VT100
Modem Connected	OFF	OFF
Supply DTR/RTS	NO	NO
Use CTR/DSR	NO	NO

Exit the RS-232 menu. Again, using the soft keys, enter the REMOTE MODE. This sets the calibrator to communicate with the DAS. The normal menu screen should disappear, and the words "Remote Mode" should appear.

▪ On-site connection of equipment to calibration system

- A manifold will be used to distribute the calibration gases from the calibrator to the various instruments. Each of the instruments will have a solenoid valve that allows ambient flow to be stopped and calibration gases to be fed to the instrument.

- All of the gases used for calibrations must be attached to the several ports on the back of the calibrator using 1/8" stainless steel tubing.

10. INSTRUMENT OR METHOD CALIBRATION

On a quarterly basis, the flow rates through the calibrator flow meters should be checked. Disconnect the calibrator from the outlet lines and attach an external flow meter so as to check the gas flow from the calibrator flow mode. The calibrator also needs to be calibrated at the end of the study. The calibrator must be sent back to the factory to be calibrated.

11. INSTRUMENT OPERATION

The calibration system will be operated in remote mode so that all commands can be executed by the data acquisition system.

12. HANDLING & PRESERVATION OF SAMPLES

Not applicable.

13. SAMPLE PREPARATION

Not applicable.

14. PREVENTIVE MAINTENANCE & REPAIRS

No preventative maintenance needs to be made on the calibrator. The calibrator should be returned to the factory for re-calibration following the 16 month field study.

15. TROUBLESHOOTING

The calibrator communications settings will be reset if the power is cycled on the instrument. Make sure that the calibrator is in REMOTE MODE or communications will not occur. The calibrator must be taken out of REMOTE MODE to use the menu driven software for on-site diagnostics. Make sure the calibrator is returned to REMOTE MODE.

Troubleshooting procedures are outlined in Section H of the manual.

16. DATA ACQUISITION, CALCULATIONS, AND DATA REDUCTION

The calibration sequences will be fully automated by the DAS via the RS-232 port.

17. COMPUTER HARDWARE & SOFTWARE

The DAS sends automated calibration sequences to the calibrator. These sequences are either self initiated by the DAS on a daily, predetermined schedule or manually initiated. The sequences are outlined below by title with specifics shown in Table 1.

DAS initiated sequences performed on a routine daily schedule

- Zero Air Reset
- NO_y zero-span calibration check with zero air, NPN, NO, and NO₂, followed by zero air
- Ozone Zero/Span calibration check with zero air and 80ppb O₃ (Angiola only).
- NO_y matrix air zero calibration check (four times daily).

Manually initiated sequences performed as specified by the site to-do list

- NO_y bi-weekly zero, span, and converter checks
 - NO_y weekly1.cal
 - NO_y weekly2.cal
 - NO_y weekly3.cal
 - NO_y weekly4.cal
- NO_y 5-point NO plus 1-pt NO₂/NO calibration
- Ozone five point calibration

Table 1. Schedule of automated and manual calibration sequences.

SCHEDULE OF AUTOMATED AND MANUAL CALIBRATION SEQUENCES								
The automated calibration sequences are listed first, followed by the manually triggered calibration sequences								
The op codes assignments are different for the NO/NOy and O3 analyzers. Keys to the op codes are as follows:								
	NO/NOy analyzer				O3 analyzer			
	0 okay, ambient sample				0 okay, ambient sample			
	1 NO span check				1 unassigned			
	2 GPT (NO2/NO) converter check				2 unassigned			
	3 NPN converter check				3 unassigned			
	4 NH3 converter check				4 O3 span check			
	5 zero air check				5 zero air check			
	6 matrix air check				6 unassigned			
	7 Instrument error				7 Instrument error			
	8 HNO3 gas				8 unassigned			
	9 Missing data				9 Missing data			
<u>Column header definitions</u>								
Time=actual time according to the data acquisition system (DAS)								
Diff. Time=the time relative to the start of the calibration sequence								
Analyzer affected=indicates the analyzer that is undergoing a calibration check								
Gas sent to analyzer=indicates the type of gas that is being delivered by the calibrator								
OP Code assigned=the assigned op code for the step, this op code is assigned until the next step is executed								
QC code assigned=incomplete								
Desired response=indicates the acceptable response from the analyzer to each step in the sequence. For the NOy analyzer, the expected NO and NOy responses are listed. These are only general guidelines.								
The following automated calibrations occur every day according to the following schedule								
Time	Diff. Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned		
Zero Air Reset								
00:10	00:00	Turn off zero air generator	NONE	NONE	NONE	NONE		
	00:01	Turn on zero air generator to activate ballast bleed timer	NONE	NONE	NONE	NONE		
NOy zero-span calibration check with zero air, NPN, NO, and NO2, followed by zero air								
Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response	
							NO	NOy
00:44	00:00	Start purging the cal lines	NONE	NONE	NONE	NONE	NONE	NONE
00:45	00:01	Start sending cal zero gas to the analyzer	NOy	zero air	5		0	0
00:46	00:02	Check the flowrates	NOy	zero air	5		0	0
00:53	00:09	Check the flowrates	NOy	zero air	5		0	0
00:55	00:11	Start sending NPN at 90 ppb to the analyzer	NOy	90ppb NPN	3		0	>70
00:56	00:12	Check the flowrates	NOy	90ppb NPN	3		0	>70

01:13	00:29	Check the flowrates	NOy	90ppb NPN	3		0	>70
01:15	00:31	Start sending NO at 90 ppb to the analyzer	NOy	90ppb NO	1		90	90
01:16	00:32	Check the flowrates	NOy	90ppb NO	1		90	90
01:26	00:42	Check the flowrates	NOy	90ppb NO	1		90	90
01:28	00:44	Start sending NO at 90 ppb, combined with 60 ppb to create 60 ppb of NO2 and 30 ppb of NO	NOy	60ppb NO2/ 30ppb NO	2		30	90
01:29	00:45	Check the flowrates	NOy	60ppb NO2/ 30ppb NO	2			
01:39	00:55	Check the flowrates	NOy	60ppb NO2/ 30ppb NO	2		30	90
01:41	00:57	Start sending zero air to the analyzer	NOy	zero air	5		0	0
01:42	00:58	Check the flowrates	NOy	zero air	5		0	0
01:49	01:05	Check the flowrates	NOy	zero air	5		0	0
01:51	01:07	End cal	NOy	NONE	NONE		NONE	NONE
01:52	01:08	Ambient recovery	NOy	NONE	NONE		NONE	NONE
02:00	01:16	Data valid again	NOy	NONE	NONE	NONE	NONE	NONE

Note: The ozone analyzer at BAC is not controlled by STI, so the following ozone zero/span check is not performed there

Ozone Zero/Span calibration check with zero air and 80ppb O3

Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response Ozone	
02:44	00:00	Start purging the cal lines	NONE	NONE	NONE	NONE	NONE	
02:45	00:01	Send zero air to O3 analyzer	O3	zero air	5		0	
02:46	00:02	Check the flowrates	O3	zero air	5		0	
02:58	00:14	Check the flowrates	O3	zero air	5		0	
03:00	00:16	Start sending 80ppb O3	O3	80ppb O3	4		80	
03:01	00:17	Check the flowrates	O3	80ppb O3	4		80	
03:10	00:26	Check the flowrates	O3	80ppb O3	4		80	
03:12	00:28	End calibration and purge cal lines	O3	NONE	NONE		NONE	
03:13	00:29	Ambient recovery	O3	NONE	NONE		NONE	
03:15	00:31	Data valid again	NONE	NONE	NONE	NONE	NONE	

NOy matrix air zero calibration check

Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response NO NOy	
05:45	00:00	Start sending matrix air to NOy	NOy	matrix air	6		0	0
06:05	00:20	End cal	NOy	NONE	NONE		NONE	NONE
06:06	00:21	Ambient recovery	NOy	NONE	NONE		NONE	NONE
06:15	00:30	Data valid again	NONE	NONE	NONE	NONE	NONE	NONE

NOy matrix air zero calibration check

Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response NO NOy	
10:45	00:00	Start sending matrix air to NOy	NOy	matrix air	6		0	0
11:05	00:20	End cal	NOy	NONE	NONE		NONE	NONE

11:06	00:21	Ambient recovery	NOy	NONE	NONE		NONE	NONE
11:15	00:30	Data valid again	NONE	NONE	NONE	NONE	NONE	NONE
NOy matrix air zero calibration check								
Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response	
							NO	NOy
15:45	00:00	Start sending matrix air to NOy	NOy	matrix air	6		0	0
16:05	00:20	End cal	NOy	NONE	NONE		NONE	NONE
16:06	00:21	Ambient recovery	NOy	NONE	NONE		NONE	NONE
16:15	00:30	Data valid again	NONE	NONE	NONE	NONE	NONE	NONE
The following calibrations are started manually and at variable times.								
NOy bi-weekly zero, span, and converter checks								
Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response	
							NO	NOy
NOy weekly1.cal								
	00:00	Start sending matrix air to NOy	NOy	matrix air	6		0	0
	00:10	Start sending zero air to NOy	NOy	zero air	5		0	0
	00:11	Check the flowrates	NOy	zero air	5		0	0
	00:18	Check the flowrates	NOy	zero air	5		0	0
	00:20	Start sending 90ppb NO	NOy	90ppb NO	1		90	90
	00:21	Check the flowrates	NOy	90ppb NO	1		90	90
	00:30	Check the flowrates	NOy	90ppb NO	1		90	90
	variable	Changing inlet filters on NOy	NOy	90ppb NO	1		90	90
NOy weekly2.cal					1			
	00:00	Start sending 90ppb NO	NOy	90ppb NO	1		90	90
	00:01	Check the flowrates	NOy	90ppb NO	1		90	90
	00:08	Check the flowrates	NOy	90ppb NO	1		90	90
	00:10	Start sending 450ppb NO	NOy	450ppb NO	1		450	450
	00:11	Check the flowrates	NOy	450ppb NO	1		450	450
	00:18	Check the flowrates	NOy	450ppb NO	1		450	450
	00:20	Start sending 450ppb NO/350ppb O3	NOy	100ppb NO 350ppb NO2	2		100	450
	00:21	Check the flowrates	NOy	100ppb NO 350ppb NO2	2		100	450
	00:28	Check the flowrates	NOy	100ppb NO 350ppb NO2	2		100	450
	00:30	Start sending zero air to NOy	NOy	zero air	5		0	0
	00:31	Check the flowrates	NOy	zero air	5		0	0
	00:38	Check the flowrates	NOy	zero air	5		0	0
	00:40	Start sending 90ppb NH3 to NOy	NOy	90ppb NH3	4		0	0
	00:41	Check the flowrates	NOy	90ppb NH3	4		0	0
	00:48	Check the flowrates	NOy	90ppb NH3	4		0	0
	00:50	Start sending zero air to NOy	NOy	zero air	5		0	0
	00:51	Check the flowrates	NOy	zero air	5		0	0
	00:55	Stop gas flows	NOy	NONE	NONE		NONE	NONE
	00:56	End cal	NONE	NONE	NONE		NONE	NONE

NOy weekly3.cal								
	00:00	Start sending HNO3 to NOy	NOy	HNO3	8		0	0
	00:01	Check the flowrates	NOy	HNO3	8		0	0
	00:13	Check the flowrates	NOy	HNO3	8		0	0
	00:15	End cal	NOy	NONE	NONE		NONE	NONE
NOy weekly4.cal								
	00:00	Start sending HNO3 to NOy	NOy	HNO3	8		0	>0
	00:01	Check the flowrates	NOy	HNO3	8		0	>0
	00:13	Check the flowrates	NOy	HNO3	8		0	>0
	00:15	Start sending zero air to NOy	NOy	zero air	5		0	0
	00:16	Check the flowrates	NOy	zero air	5		0	0
	00:23	Check the flowrates	NOy	zero air	5		0	0
	00:25	Start sending matrix air to NOy	NOy	matrix air	6		0	0
	00:35	End cal	NOy	NONE	NONE		NONE	NONE
	00:36	Ambient recovery	NOy	NONE	NONE		NONE	NONE
	00:44	Data valid again	NOy	NONE	NONE	NONE	NONE	NONE
NOy 5-point NO plus 1-pt NO2/NO calibration								
Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response	
							NO	NOy
	00:00	Purge the cal lines	NONE	NONE	NONE	NONE	NONE	NONE
	00:01	Start sending zero air to NOy	NOy	zero air	5		0	0
	00:02	Check the flowrates	NOy	zero air	5		0	0
	00:19	Check the flowrates	NOy	zero air	5		0	0
	00:21	Start sending 450ppb NO to NOy	NOy	450ppb NO	1		450	450
	00:22	Check the flowrates	NOy	450ppb NO	1		450	450
	00:39	Check the flowrates	NOy	450ppb NO	1		450	450
	00:41	Start sending 350ppb NO to NOy	NOy	350ppb NO	1		350	350
	00:42	Check the flowrates	NOy	350ppb NO	1		350	350
	00:59	Check the flowrates	NOy	350ppb NO	1		350	350
	01:01	Start sending 250ppb NO to NOy	NOy	250ppb NO	1		250	250
	01:02	Check the flowrates	NOy	250ppb NO	1		250	250
	01:19	Check the flowrates	NOy	250ppb NO	1		250	250
	01:21	Start sending 150ppb NO to NOy	NOy	150ppb NO	1		150	150
	01:22	Check the flowrates	NOy	150ppb NO	1		150	150
	01:39	Check the flowrates	NOy	150ppb NO	1		150	150
	01:41	Start sending 75ppb NO to NOy	NOy	75ppb NO	1		75	75
	01:42	Check the flowrates	NOy	75ppb NO	1		75	75
	01:59	Check the flowrates	NOy	75ppb NO	1		75	75
	02:01	Start sending zero air to NOy	NOy	zero air	5		0	0
	02:02	Check the flowrates	NOy	zero air	5		0	0
	02:19	Check the flowrates	NOy	zero air	5		0	0
	02:21	Start sending 450ppb NO/350 ppb O3	NOy	350ppb NO2 100 ppb NO	2		100	350
	02:22	Check the flowrates	NOy	350ppb NO2 100 ppb NO	2		100	350
	02:39	Check the flowrates	NOy	350ppb NO2 100 ppb NO	2		100	350
	02:41	Start sending zero air to NOy	NOy	zero air	5		0	0
	02:42	Check the flowrates	NOy	zero air	5		0	0
	02:59	Check the flowrates	NOy	zero air	5		0	0
	03:01	End cal	NOy	NONE	NONE		NONE	NONE

	03:02	Ambient recovery	NOy	NONE	NONE		NONE	NONE
	03:07	Data valid again	NOy	NONE	NONE	NONE	NONE	NONE
Ozone five point calibration-performed on command only, not automatic								
Time	Diff Time	Comments	Analyzer Affected	Gas sent to analyzer	OP Code Assigned	QC Code Assigned	Desired Response Ozone	
	00:00	Purge the calibration lines	NONE	NONE	NONE	NONE	NONE	
	00:01	Start sending zero air to O3	O3	zero air	5		0	
	00:02	Check actual flowrates	O3	zero air	5		0	
	00:19	Check actual flowrates	O3	zero air	5		0	
	00:21	Start sending 240ppb O3 to O3	O3	240ppb O3	4		240	
	00:22	Check actual flowrates	O3	240ppb O3	4		240	
	00:34	Check actual flowrates	O3	240ppb O3	4		240	
	00:36	Start sending 180ppb O3 to O3	O3	180ppb O3	4		180	
	00:37	Check actual flowrates	O3	180ppb O3	4		180	
	00:49	Check actual flowrates	O3	180ppb O3	4		180	
	00:51	Start sending 120ppb O3 to O3	O3	120ppb O3	4		120	
	00:52	Check actual flowrates	O3	120ppb O3	4		120	
	01:04	Check actual flowrates	O3	120ppb O3	4		120	
	01:06	Start sending 60ppb O3 to O3	O3	60ppb O3	4		60	
	01:07	Check actual flowrates	O3	60ppb O3	4		60	
	01:19	Check actual flowrates	O3	60ppb O3	4		60	
	01:21	End cal	O3	NONE	NONE		NONE	
	01:22	Ambient recovery	O3	NONE	NONE		NONE	
	01:23	Data valid again	O3	NONE	NONE	NONE	NONE	

18. DATA MANAGEMENT & RECORDS MANAGEMENT

See QIWP.

Calibrator targeted gas concentrations are recorded by the DAS. See the DAS SOP for further details.

A logbook must be kept to record any problems encountered, maintenance performed, or major repairs.

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